

# PATENT SPECIFICATION

NO DRAWINGS

Inventor: JAMES ALBERT MOYSE

1,154,730

1,154,730



Date of filing Complete Specification: 21 Sept., 1966.

Application Date: 8 Oct., 1965.

No. 42782/65.

Complete Specification Published: 11 June, 1969.

© Crown Copyright 1969.

Index at acceptance: —D1 P(1A3, 1A4, 1C1A, 1C2B, 1C4, 1CX, 1D); C5 D(6A4A, 6B7, 6B12B1, 6B12F1, 6B12G6, 6B12K2, 6B12N1, 6B12N2, 6B12N3, 6B12N4, 6B13, 6C8)

Int. Cl.: —D 0611/12

## COMPLETE SPECIFICATION

### Improvements in the Laundering of Synthetic Polymeric Textile Materials

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED of Imperial Chemical House, Millbank, London, S.W.1, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to a process for minimising soil redeposition during the laundering of textiles containing more than 50% by weight of synthetic polymeric materials.

During laundering operations, the soiling matter present on the textile materials being washed is removed and becomes suspended in the laundering liquor; part of this soiling matter will be retained in the liquor, which is subsequently discarded, but part of it is normally redeposited on the textile materials and is not readily removed therefrom by rinsing. For this reason the efficiency of laundering operations in removing soiling matter is limited, and it is possible, when lightly soiled textile materials are washed together with those that are more heavily soiled, for the former to emerge from laundering in an equally soiled or even a more soiled condition than that which was the case before laundering.

Textiles which are composed wholly or substantially of synthetic polymeric materials are particularly prone to become soiled by this redeposition of soil during laundering operations.

According to the present invention there is provided a process for reducing the extent to which soil redeposition occurs during the laundering of textiles comprising more than

50% by weight of synthetic polymeric materials which comprises carrying out the laundering operation with an aqueous composition comprising, in addition to a detergent, a polycondensate containing either ester repeating groups or amide repeating groups, but not both, and also at least one active group, the said active group or groups being selected from acidic groups, salts of acidic groups and water-soluble polymeric groups as hereinafter defined.

Acidic groups and their salts which may function as the active group of groups in the polycondensates used according to the invention include both simple and polymeric acidic groups and their salts, and they may be present in the polycondensate molecule as end-groups or they may be attached to other groups at more than one position. The acidic groups or salts thereof may be relatively weak acids or salts thereof, such as carboxylic acids or carboxylic acid salts, or they may be relatively strong acids or salts thereof, such as sulphonic or phosphoric acids or salts of such acids. As examples of simple or monomeric acidic groups which may be present in the polycondensate molecule there may be mentioned acidic end-groups such as *p*-sulphobenzoic ester groups and acidic difunctional groups such as 5-sulphoisophthalic ester groups. As an example of polymeric acidic groups there may be mentioned poly-(ethylene-5-sulpho-isophthalate) groups.

Water-soluble polymeric groups which may function as the active group or groups in the polycondensates used according to the invention are polymeric groups derived from water-soluble polymers having an average molecular weight of at least 100. Examples

[Price 4s. 6d.]

of water-soluble polymeric groups are groups derived from poly(vinylalcohol), poly(vinyl methyl ether), poly (N,N-dimethylacrylamide), methyl cellulose, hydroxyethyl cellulose and water-soluble poly(oxyalkylene) glycols.

The polycondensates which are preferred for use in the process of the invention are those in which the active group or groups are polyoxyalkylene groups. Suitable polyoxyalkylene groups include polyoxyethylene, polyoxypropylene, polyoxytrimethylene, polyoxetramethylene and polyoxybutylene and copolymers thereof. Particularly preferred polyoxyethylenes are those containing polyoxyethylene groups.

The active groups present in the polycondensates may all be of the same type or they may be of different types. Thus, for example, the polycondensate may contain both acidic active groups and water-soluble polymeric groups; furthermore, the acidic groups may be of more than one species, for instance both sulphonic acid and phosphoric acid groups may be present, and similarly the water-soluble polymeric groups may be of more than one species, such as both polyoxyethylene and polyoxypropylene groups.

The polycondensates used in the process of the invention may be obtained from the appropriate starting materials by any of the known methods for preparing condensation polymers. As already stated, the polycondensates are of the type in which the repeating groups are either ester groups or amide groups, but not both. Polycondensates containing ester repeating groups may be obtained, for example, by condensing together dihydric alcohols with dicarboxylic acids or with suitable derivatives of such acids, for example the lower alkyl esters. Polycondensates containing amide repeating groups may be obtained, for example, by self-condensation of a lactam, or by condensation of a dicarboxylic acid with a diamine or an amino-acid or lactam. The active groups may be introduced into the polycondensates during the course of the main condensation reaction; this may be achieved by utilising as starting materials compounds containing the desired active groups in addition to the ester- or amide-forming functional groups referred to above. Thus, for example, sulphonic acid active groups may be introduced into a polycondensate containing either ester or amide groups by using as one of the starting materials a sulphocarboxylic acid, and polyoxyalkylene groups may be introduced by using as one of the starting materials a polyoxyalkylene compound having one or more hydroxyl end groups or one or more amino end-groups. Alternatively, the active groups may be introduced into the polycondensates in a second step following the main condensation reaction; for example, a simple polyamide containing reactive amide

groups may be reacted with an alkylene oxide to produce polyoxyalkylene groups attached to the main polymer chain. Condensation of the starting materials referred to above may be affected under the conditions well known for carrying out such reactions. Thus the starting materials may conveniently be mixed and heated, for example within the range 150°C to 300°C for a period of from 30 minutes to 12 hours. It may be advantageous to carry out the condensation in an atmosphere of an inert gas such as nitrogen, or it may in some cases be preferable to incorporate a solvent or flux in the reaction mixture. Catalysts for the condensation reaction may be incorporated if desired, together with antioxidants or stabilisers to prevent decomposition of the reactants. It may frequently be advantageous to carry out the final stages of the condensation at a reduced pressure.

As examples of particular types of polycondensate which are valuable in the process of the present invention, there may be mentioned the polyester condensates containing acidic or water-soluble groups which are described in British Patent Specification No. 1,088,984, in particular the polyester condensates therein described which contain polyoxyethylene groups, and the polyamide condensates containing polyoxyalkylene groups which are described in British Patent Specification Nos. 1,108,811 and 1,108,812.

The textile materials to which the process of the present invention may be applied are those comprising more than 50% by weight of synthetic polymeric materials such as polyolefines, for example polyethylene and polypropylene, polyamides, for example poly(hexamethylene adipamide) and polymers of caprolactam, polyesters, for example poly(ethylene terephthalate), polymers and copolymers of acrylonitrile, cellulose esters, for example cellulose triacetate, and glass, that is to say textile materials which are composed wholly of such synthetic polymeric materials or are composed of blends of such materials with minor proportions of natural polymeric materials such as cellulosic and proteinaceous materials.

The textile materials to which the process of the invention is applied are normally in the form of fabrics comprising made-up articles and garments.

In carrying out the process of the invention, the polycondensates hereinbefore described are added directly to the laundering liquor. In most cases it is convenient to add the polycondensate in the form of a solution or dispersion in water or in a suitable organic solvent or in a mixture of such a solvent with water. The solubility of the polycondensates in water varies, but those polycondensates which do not readily dissolve or disperse in water may be dispersed by known methods, such as ball- or gravel-

milling. The amount of polycondensate added to the laundering liquor is normally from 0.0001% to 1.0% of the weight of the liquor, although larger amounts than this may be employed if desired. Instead of a single polycondensate, a mixture of two or more polycondensates as hereinbefore described may be used.

- The polycondensates may be used in the process of the invention in conjunction with any of the known types of anionic, non-ionic and cationic detergents normally used in laundering operations. These include the anionic detergents such as the sulphates, sulphonates and carboxylic acid salts derived from natural fatty materials and from petroleum and related raw materials; the non-ionic detergents such as the polyethenoxy derivatives of alcohols and acids available from natural and synthetic sources, of fatty amides and amines and of synthetic phenols and mercaptans; and the cationic detergents such as amine salt and quaternary ammonium salts based on both natural and synthetic raw materials. The laundering liquor may contain, in addition to the detergent, any of the other normal laundering adjuvants such as alkalies, bleaching agents, sequestering agents, fluorescent brightening agents, cellulose or starch ethers and esters, and inorganic salts.

- If desired, the polycondensate may be added to the laundering liquor in the form of a dispersion in a solution of the detergent, or it may even be compounded with the solid detergent, optionally together with one or more of the adjuvants mentioned above, to form a single composition which may be added directly to the laundering liquor.

- The invention is illustrated but not limited by the following Examples, in which percentages and ratios are by weight:—

#### EXAMPLE 1

- A piece of polyester fibre fabric was laundered in the presence of a piece of cotton fabric four times the weight of the polyester fabric, which had been synthetically soiled with a composition comprising lampblack, tallow, paraffin and trichloroethylene, using a laundering liquor containing 0.1% of sodium cetyl sulphate, 0.1% of soda ash and an aqueous non-ionic dispersion of 0.025% of the copolymer of polyethylene terephthalate and polyoxyethylene glycol made as described in Example 11 of British Specification No. 1,088,984. The liquor to goods ratio was 33:1. Laundering was carried out with agitation at a temperature of 80°C for 15 minutes, followed by four rinses in warm water.

- A similar treatment was carried out in which the copolymer of polyethylene terephthalate and polyoxyethylene glycol was omitted from the laundering liquor.

The polyester fibre fabric which was

laundered in the presence of the copolymer of polyethylene terephthalate and polyoxyethylene glycol was found to be much less soiled than that which had been laundered in the liquor from which the copolymer had been omitted, indicating that redeposition of soil on the polyester fabric had been largely prevented.

#### EXAMPLE 2

When, in Example 1, the polyester fabric was replaced by a polyester/cotton fabric, comprising 67% polyester and 33% cotton, of one third of the weight of the synthetically soiled cotton fabric, much less soil was re-deposited from the soiled cotton on to the polyester/cotton fabric when the co-polymer of polyethylene terephthalate and polyoxyethylene glycol was present in the laundering liquor than when it was omitted.

#### EXAMPLE 3

A piece of a polyamide fibre fabric was laundered in the presence of a synthetically soiled piece of cotton fabric, prepared as described in Example 1 and one and a half times the weight of the polyamide fabric, using a liquor containing 0.1% "Persil" (Regd. Trade Mark) and 0.2% of a 12.4% aqueous non-ionic dispersion of the copolymer of  $\alpha,\omega$ -diaminopolyoxyethylene of mol. wt. 1532, dimethyl terephthalate and caprolactam made as described in Example 12 of British Patent Specification No. 1,108,812. The liquor to goods ratio was 25:1. Laundering was carried out at a temperature of 80°C for 15 minutes, followed by rinsing in warm water. A control treatment was also carried out in which the copolymer was omitted from the laundering liquor.

The polyamide fibre fabric which was laundered in the presence of the copolymer of  $\alpha,\omega$ -diamino polyoxyethylene, dimethyl terephthalate and caprolactam was much cleaner than that which had been laundered in the liquor from which the copolymer had been omitted.

#### EXAMPLE 4

A piece of a polyacrylonitrile fibre fabric was laundered in the presence of a synthetically soiled piece of cotton fabric, prepared as described in Example 1 and twice the weight of the polyacrylonitrile fabric, using a liquor containing 0.1% "Tide" (Regd. Trade Mark) and an aqueous dispersion of 0.1% of the copolymer of polyethylene terephthalate and polyoxyethylene glycol made as described in Example 11 of British Patent Specification No. 1,088,984. The liquor to goods ratio was 33:1. Laundering was carried out with agitation at a temperature of 65°C for 15 minutes, followed by four rinses in warm water. A control laundering treatment was carried out in which the copolymer was omitted from the laundering liquor.

The polyacrylonitrile fabric laundered in the presence of the copolymer was found to be soiled to a lesser degree than that which had been laundered in the liquor from which the copolymer had been omitted.

#### EXAMPLE 5

A piece of a polyester fibre fabric was laundered in the presence of a piece of a synthetically soiled piece of cotton fabric, prepared as described in Example 1 and four times the weight of the polyester fabric, in a liquor containing 0.1% "Persil" (Regd. Trade Mark) and 0.1% of a 12.4% aqueous dispersion of the copolymer of  $\alpha,\omega$ -diaminopolyoxyethylene of mol. wt. 1432, dimethyl terephthalate and caprolactam made as described in Example 12 of British Patent Specification No. 1,108,812. The liquor to goods ratio was 33:1. Laundering was carried out at 80°C for 15 minutes followed by four rinses in warm water. A control treatment was carried out in which the copolymer was omitted from the laundering liquor.

The polyester fabric which was washed in the presence of the copolymer was much cleaner than that which had been laundered in the liquor from which the copolymer had been omitted.

#### EXAMPLE 6

A piece of a polyester fibre fabric was laundered in the presence of a piece of synthetically soiled cotton fabric, prepared as described in Example 1 and four times the weight of the polyester fabric, using a liquor containing 0.1% cetyl sulphate, 0.1% soda ash and an aqueous dispersion of 0.025% of the sulphonated copolymer made as described in Example 2 of British Patent Specification No. 1,127,925. The liquor to goods ratio was 30:1. Laundering was carried out at a temperature of 80°C. for 15 minutes, followed by four rinses in warm water. A similar treatment was carried out in which the sulphonated copolymer was omitted from the laundering liquor.

The polyester fabric laundered in the presence of the copolymer was found to be soiled to a lesser extent than that which had been laundered in the liquor from which the copolymer had been omitted.

#### EXAMPLE 7

When, in Example 5, the polyester fabric was replaced by a 67% polyester/33% cotton fabric, of one third of the weight of the synthetically soiled cotton fabric, considerably less soil was redeposited on the polyester/cotton fabric from the laundering liquor containing the copolymer of  $\alpha,\omega$ -diaminopolyoxyethylene, dimethyl terephthalate and caprolactam than from the liquor to which the copolymer had not been added.

#### EXAMPLE 8

A piece of a polyester fibre fabric was laundered in the presence of a piece of synthetically soiled cotton fabric, prepared as described in Example 1 and twice the weight of the polyester fabric, using a liquor containing 0.1% of soap, 0.12 part of a 20% aqueous anionic dispersion of the triazinyl fluorescent brightening agent made as described in Example 15 of British Patent Specification No. 985,484, and 0.2% of a 15% aqueous non-ionic dispersion of the copolymer of polyethylene terephthalate and polyoxyethylene glycol made as described in Example 11 of British Patent Specification No. 1,088,984. The liquor to goods ratio was 50:1. Laundering was carried out with agitation at a temperature of 80°C for 15 minutes, followed by four rinses in warm water.

A similar treatment was carried out in which the copolymer of polyethylene terephthalate and polyoxyethylene glycol was omitted from the laundering liquor.

The polyester fibre fabric was laundered in the presence of the copolymer of polyethylene terephthalate and polyoxyethylene glycol was found to be much less soiled and much whiter in appearance than that which had been laundered in the liquor from which the copolymer had been omitted.

#### WHAT WE CLAIM IS:—

1. A process for reducing the extent to which soil redeposition occurs during the laundering of textiles comprising more than 50% by weight of synthetic polymeric materials which comprises carrying out the laundering operation with an aqueous composition comprising, in addition to a detergent, a polycondensate containing either ester repeating groups or amide repeating groups, but not both, and also at least one active group, the said active group or groups being selected from acidic groups, salts of acidic groups and water-soluble polymeric groups as hereinbefore defined.

2. A process as claimed in claim 1, wherein the active group present in the polycondensate is a polyoxyalkylene group.

3. A process as claimed in claim 2, wherein the polyoxyalkylene group present in the polycondensate is a polyoxyethylene group.

4. A process as claimed in claim 1, wherein the active group present in the polycondensate is a sulphonic acid group or salt thereof.

5. A process as claimed in claim 1, wherein the polycondensate contains active groups of more than one of the types therein specified.

6. A process as claimed in claim 1, wherein the active groups include groups of more than one species of a type of group therein specified.

7. A process as claimed in any one of claims 1 to 6, wherein the textile is composed of synthetic polyester fibres.

8. A process as claimed in any one of claims 1 to 6, wherein the textile is composed of a blend of synthetic polyester fibres and cellulose fibres.
- 5 9. A process as claimed in any one of claims 1 to 6, wherein the textile is composed of synthetic polyamide fibres.
- 10 10. A process as claimed in any one of claims 1 to 6, wherein the textile is composed of polyacrylonitrile fibres.
11. A process as claimed in any one of claims 1 to 10, wherein the polycondensate is added to the laundering liquor in the form of an aqueous solution or dispersion.
12. A process as claimed in any one of claims 1 to 11, wherein the amount of the polycondensate added to the laundering liquor is from 0.0001% to 1.0% of the weight of the liquor.
13. A process as claimed in any one of claims 1 to 12, wherein more than one polycondensate as defined in claim 1 is employed.
14. A process according to claim 1 substantially as hereinbefore described, with particular reference to the foregoing Examples.

WATER SCOTT,  
Agent for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1969.

Published by the Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.